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Indian Standard

IS 1448 (Part 18) : 2020

पेट्रोलियम एवं इसके उत्पादों की परीक्षण पद्धतियाँ

भाग 18 पेट्रोलियम उत्पादों का आसवन

(तीसरा पुनरीक्षण)

Methods of Test for Petroleum and its Products

Part 18 Distillation of Petroleum Products

(Third Revision)

ICS 75.080

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Methods of Sampling and Test for Petroleum, its Products, Gaseous Fuels and Lubricants Sectional Committee, PCD 01

FOREWORD

This Indian Standard (Part 18) (Third Revision) was adopted by the Bureau of Indian Standards on recommendation of the Methods of Sampling and Test for Petroleum, its Products, Gaseous Fuels and Lubricants Sectional Committee and approval of the Petroleum, Coal and Related Products Division Council.

The second revision of this standard was published in 1991 and was adoption of ASTMD 86-82 and IP 123/84.

This third revision is carried out as the test procedure has been improved by ASTM and IP. In this revision, considerable assistance has been derived from ASTM D 86 – 2015 and IP123/2014. This standard is modified mainly to extend the scope to include motor gasoline-ethanol fuel blends with greater than 10 percent volume ethanol and diesel-biodiesel blends upto 20 percent biodiesel. This standard covers a laboratory test method for the atmospheric non fractionating distillation, utilizing either manual or automated equipment.

The use of this document can involve hazardous materials, operations and equipment. This Standard does not purport to address all of the safety problems associated with its use. It is the responsibility of users of this document to take appropriate measures to ensure the safety and health of personnel prior to the application of the standard, and to determine the applicability of regulatory limitations and any other restrictions for this purpose.

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2: 1960 ‘Rules for rounding off numerical values (*revised*)’.

Indian Standard

METHOD OF TEST FOR PETROLEUM AND ITS PRODUCTS

Part 18 DISTILLATION OF PETROLEUM PRODUCTS

(Third Revision)

1 SCOPE

This method of test covers the distillation of motor gasoline, ethanol fuel blends with greater than 10 percent volume ethanol, diesel fuels, biodiesel blends upto 20 percent biodiesel, aviation gasoline, aviation turbine fuels, special boiling point spirits, napthas, white spirit, kerosene, gas oils, distillate fuel oils and similar petroleum products.

NOTE — This method is not intended for the distillation of natural gasoline and volatile organic liquids or relatively pure solvents.

This method of test covers a laboratory test method for the atmospheric distillation, utilizing either manual or automated equipment, to determine the boiling range of light and middle distillates derived from petroleum and having initial boiling points (IBP) above 0°C and end points below approximately 400°C, automotive spark ignition engine fuels with or without oxygenates, burner fuels and marine fuels that have no appreciable quantities of residue.

2 REFERENCES

The following standards contain provisions which, through reference in this text, constitute provisions of this standard. At the time of publication the editions indicated were valid. All standards are subject to revision, and parties to agreements based on the standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
1447 (Part 1) : 2000	Petroleum and its products — Methods of sampling: Part 1 Manual sampling (*first revision*)

3 SUMMARY OF THE METHOD

The sample is placed in one of the four groups, based on its composition, vapour pressure, expected initial boiling point (IBP) or expected final boiling point (FBP). The apparatus arrangement, condenser temperature and other operational variables are defined by the group in which the sample falls. A 100 ml test portion is distilled under the specified conditions appropriate to the group into which the sample falls, and systematic observations of thermometer readings and volumes of condensate recovered are made. The volume of the residue in the flask is measured, and the loss on distillation recorded. The thermometer readings are corrected for barometric pressure and from the data, the results of the test are calculated and reported as per specification requirement.

Test results are commonly expressed as percent evaporated or percent recovered *versus* corresponding temperature, either in a table or graphically, as a plot of the distillation curve.

4 SIGNIFICANCE

The boiling range gives information on the composition, the properties, and the behaviour of the fuel during storage and use. Thus the distillation that is, volatility characteristics of hydrocarbon have an important effect on their safety and performance. Volatility is the major determinant of the tendency of a hydrocarbon mixture to produce potentially explosive vapours.

The distillation characteristics are critically important for both automotive and aviation gasolines, affecting starting, warm-up, and tendency to vapour lock at high operating temperature or at high altitude, or both. The presence of high boiling point components in these and other fuels can significantly affect the degree of formation of solid combustion deposits.

Volatility, as it affects rate of evaporation, is an important factor in the application of many solvents, particularly those used in paints.

5 TERMINOLOGY

For the purposes of this standard, the following terms and definitions shall apply.

5.1 Initial Boiling Point — The thermometer reading (corrected) that is, observed instant at the first drop of condensate falls from the lower end of the condenser tube.

5.2 End Point or Final Boiling Point — The maximum thermometer reading (corrected) obtained during the test.

5.3 Dry Point — The thermometer reading (corrected) that is observed at the instant the last drop of liquid evaporates from the lowest point in the flask, any drops or film of liquid on the side of the flask or on the thermometer being disregarded.

NOTE — The end point (final boiling point), rather than the dry point, is intended for general use. The dry point can be reported in connection with special purpose naphtha, such as those used in the paint industry.

5.4 Decomposition Point — The thermometer reading which coincides with the first indication of thermal decomposition of the liquid in the flask.

NOTE — Characteristic indications of thermal decomposition are an evolution of fumes, and erratic thermometer readings which usually show a decided decrease after any attempt is made to adjust the heat.

5.5 Percent Recovered — The volume of condensate, in millilitres, observed in the receiving cylinder at any point in the distillation in connection with a simultaneous temperature reading.

NOTE — It is expressed as a percentage of the charge volume.

5.6 Percent Recovery — The maximum percent recovered, relative to the sample charge.

5.7 Percent Total Recovery — The combined percent recovery and percent residue in the flask.

5.8 Percent Loss — 100 minus the percent total recovery.

5.9 Percent Residue — The percent total recovery minus the percent recovery, or the volume of residue, in millilitres, if measured directly.

5.10 Percent Evaporated — The sum of the percent recovered and the percent loss.

5.11 Temperature Lag, n — The offset between the temperature reading obtained by a temperature sensing device and the true temperature at that time.

6 APPARATUS

6.1 General

6.1.1 The basic components of the distillation unit are the distillation flask, the condenser and associated cooling bath, a metal shield or enclosure for distillation flask, the heat source, the flask support, the temperature measuring device and the receiving cylinder to collect the distillate.

6.1.2 Typical assemblies of the manual apparatus are shown in Fig. 1 and 2.

6.1.3 In addition to the basic components described in this clause, automated apparatus also are equipped with a system for measuring and automatically recording the vapour temperature and the associated recovered volume in the receiving cylinder.

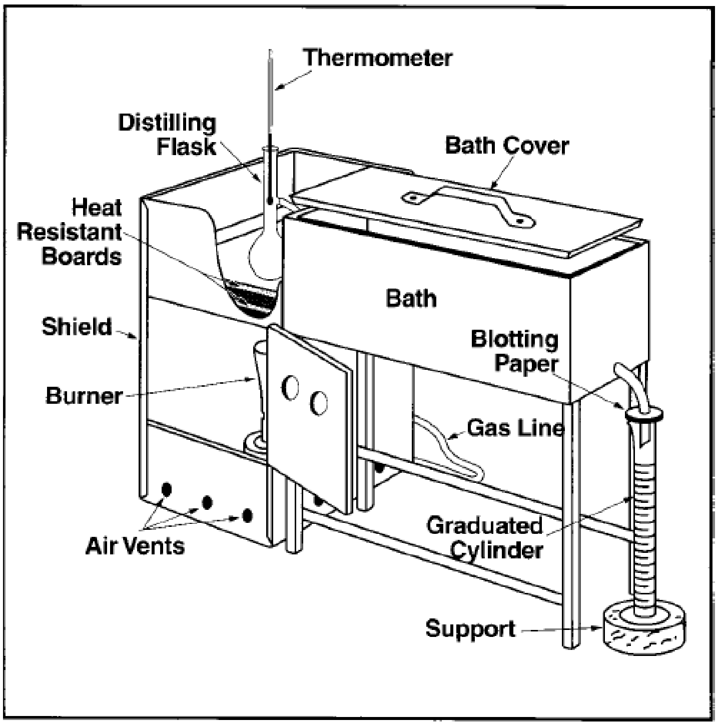
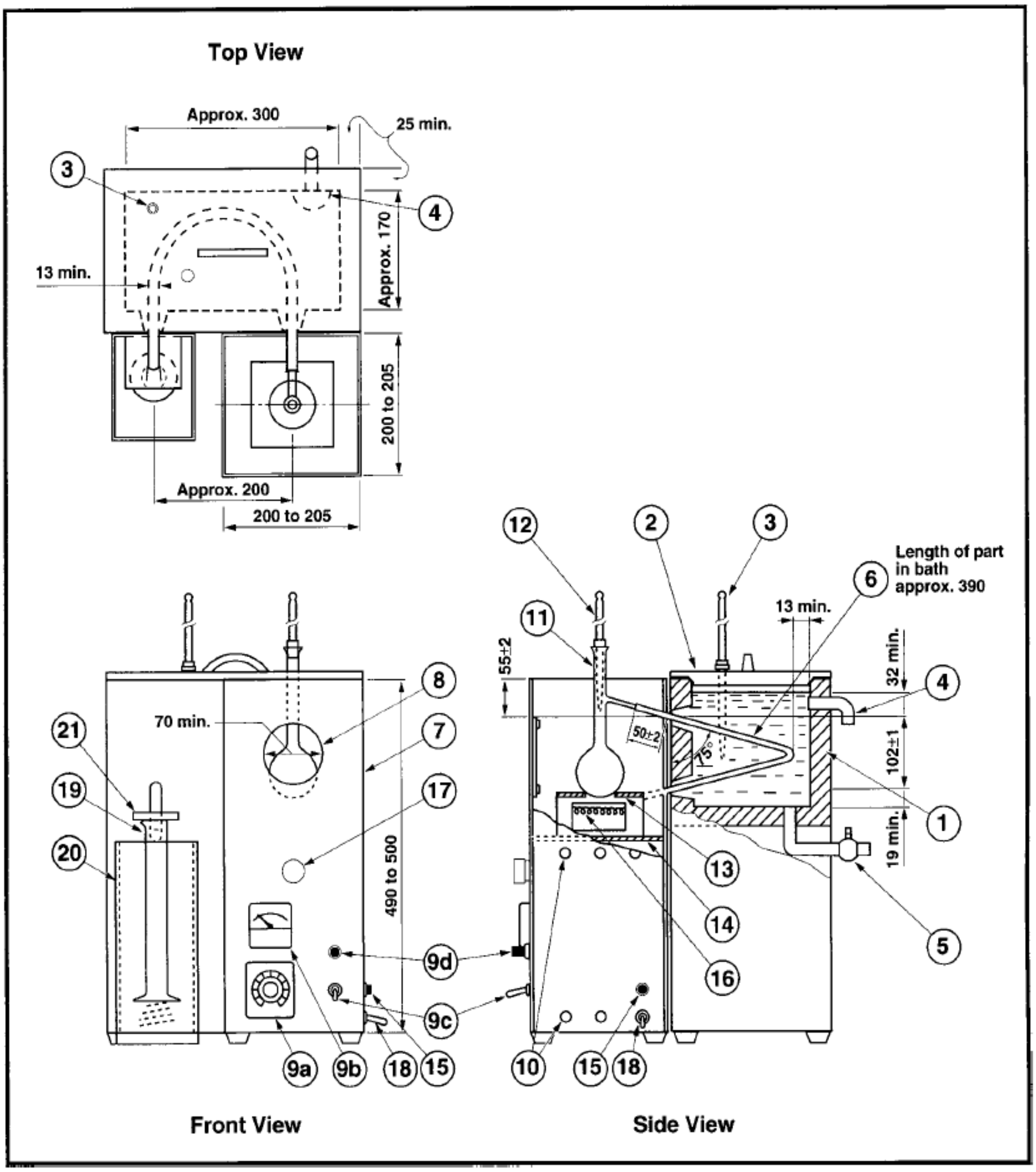


FIG. 1 APPARATUS ASSEMBLY USING GAS BURNER



1–Condenser bath
2–Bath cover
3–Bath temperature sensor
4–Bath overflow
5–Bath drain
6–Condenser tube
7–Shield
8–Viewing window
9a–Voltage regulator
9b–Voltmeter or ammeter
9c–Power switch
9d–Power light indicator
10–Vent
11–Distillation flask
12–Temperature sensor
13–Flask support board
14–Flask support platform
15–Ground connection
16–Electric heater
17–Knob for adjusting level of support platform
18–Power source cord
19–Receiver cylinder
20–Receiver cooling bath
21–Receiver cover

Fig. 2 Apparatus Assembly Using Electric Heater

6.2 Distillation Flasks

The distillation flasks shall have a capacity of 125 ml and be constructed of heat-resistant glass, according to the dimensions and tolerances shown in Fig. 3 and 4**.**

NOTE — For tests specifying the dry point, specially selected flasks with bottoms and walls of uniform thickness are desirable.

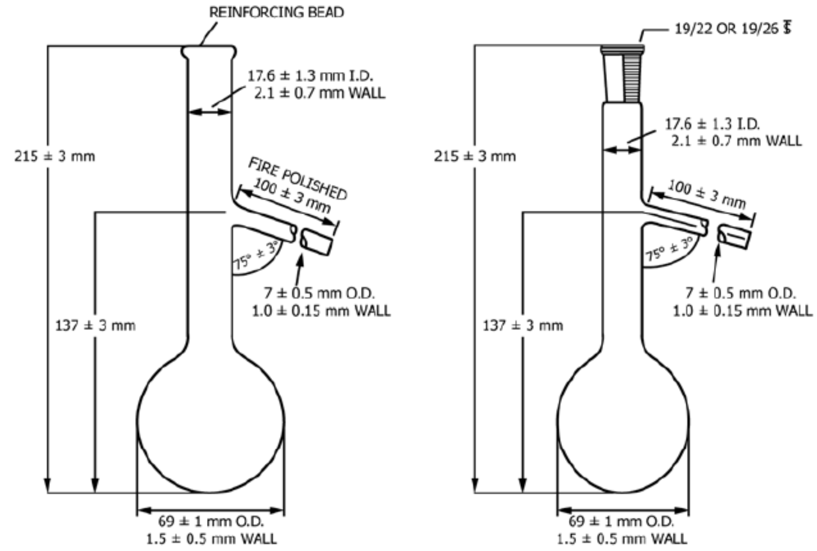
6.3 Condenser Tube and Cooling Bath

6.3.1 Typical types of condenser and cooling bath are illustrated in Fig. 1 and 2.

6.3.2 The condenser shall be made of seamless non-corrosive metal tubing, 560 mm ± 5 mm in length, with an outside diameter of 14 mm and a wall thickness of 0.8 mm to 0.9 mm.

NOTE — Brass or stainless steel are suitable materials.

6.3.3 The condenser shall be set so that a length of 393 mm ± 3 mm of the tube is in contact with the cooling medium, with 50 mm ± 3 mm outside the cooling bath at the upper end, and 114 mm ± 3 mm outside at the lower end. The portion of tube projecting at the upper end shall be set at an angle of 75° ± 3 with the vertical. The portion of the tube inside the cooling bath shall be either straight or bent in any suitable continuous smooth curve. The average gradient shall be 15° ± 1° with respect to the horizontal, and no 100 mm section shall have a gradient outside the range of 15° ± 3°. The projecting lower portion of the condenser tube shall be curved downward for a length of 76 mm and the lower end cut off at an acute angle. Provisions shall be made to enable the flow of distillate to run down the side of the receiving cylinder. Figure 5 gives an illustration of the lower end of the condenser tube.



125 mL Flask **125 mL Flask with Ground Glass Joint**

Fig. 3 125 ML Flask and 125 ML Flask with Ground Glass Joint

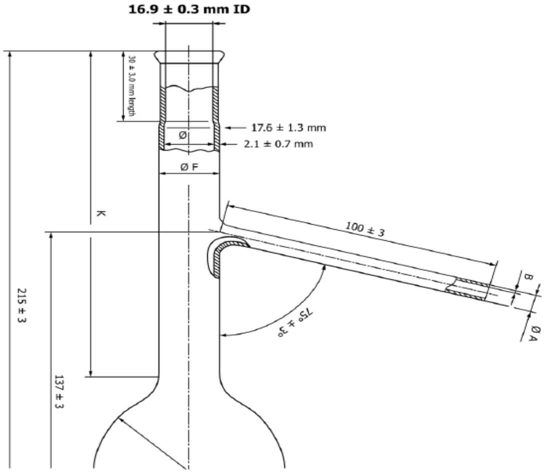


Fig. 4 Details of Upper Neck Section

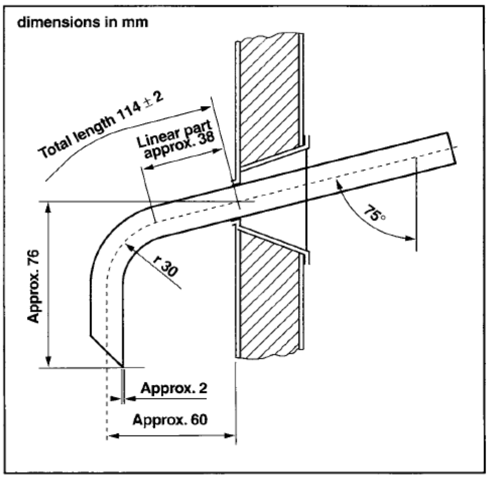


Fig. 5 Lower End of Condenser Tube

The flow of distillate down the side of the graduated cylinder may be accomplished either by using a drip deflector inserted in the receiver, or by having the downward length of the condenser tube curve slightly backwards so as to ensure contact with the wall of the receiving cylinder at a point 25 mm to 32 mm below the top of the receiving cylinder when it is in position to receive distillate.

6.3.4 The volume and design of the cooling bath will depend on the cooling medium employed. The cooling capacity of the bath shall be adequate to maintain the required temperature for the desired condenser performance. A single cooling bath may be used for several condenser tubes.

6.4 Metal Shield or Enclosure for Flask (Manual Apparatus Only)

Shields shall be provided to protect the operator from damage to the unit during operation, and to protect the distillation flask from draughts. These shall allow easy access to the distillation setup during operation, and be provided with at least one window for observing the dry point at the end of the distillation.

NOTES:

1 A typical shield for a unit fitted with a gas burner would be 480 mm high, 280 mm long and 200 mm wide, made of sheet metal approximately 0,8 mm in thickness (*see* Fig. 1).

2 A typical shield for a unit fitted with an electric heater would be 440 mm high, 200 mm long and 200 mm wide, made of sheet metal approximately 0,8 mm in thickness (*see* Fig. 2).

6.5 Heat Source

6.5.1 *Gas Burner* (*see Fig. 1)*

Capable of bringing over the first drop from a cold start within the time specified, and of continuing the distillation at the specified rate. A sensitive regulating valve and gas pressure regulator to give complete control of heating shall be provided.

6.5.2 *Electric Heater* (*see Fig. 2)*

Of low heat retention and adjustable from 0W to 1 000 W.

6.6 Flask Support

6.6.1 *Type 1, for Use with Gas Burner* (*see Fig. 1)*

Either a ring support of the ordinary laboratory type, 100 mm or larger in diameter, supported on a stand inside the shield, or a platform adjustable from the outside of the shield shall be used. On this ring or platform is mounted a hard board made of ceramic or other heat resistant material, 3 mm to 6 mm in thickness with a central opening 76 mm to 100 mm in diameter and outside line dimensions slightly smaller than the inside boundaries of the shield. The flask support board shall be constructed of ceramic or other heat resistant material, 3 mm to 6 mm in thickness and shall have a central opening conforming to the dimensions given in Table 3. The flask support board shall be of sufficient dimension to ensure that thermal heat to the flask only comes from the central opening and that extraneous heat to the flask other than through the central opening is minimized. The flask support board may be moved slightly in accordance with the directions for positioning the distillation flask so that direct heat is applied to the flask only through the opening in this board. The position of the flask is set by adjusting the length of the side arm inserted into the condenser.

6.6.2 *Type 2, for Use with an Electric Heater* (*see Fig. 2*)

The flask support is a platform on top of the electric heater, adjustable from outside of the shield. The flask support board described in **6.6.1** is mounted on this support. Provision shall be made for moving the upper (flask support) board slightly in the horizontal plane to ensure that direct heat is applied only through the specified opening in this board. The flask support assembly shall be able to move vertically to ensure contact of the flask support board with the bottom of the distillation flask during the distillation, and to allow for easy mounting and removal of the distillation flask from the unit.

6.7 Graduated Cylinders

6.7.1 *Receiving Cylinder*

Of 100 ml ± 1.0 ml capacity. It shall be graduated at intervals of 1 ml beginning at least at the 5 ml mark and have a graduation at the 100 ml mark. The shape of the base shall be such that the receiver does not topple when placed empty on a surface inclined at an angle of 13° to the horizontal. Construction details and tolerances for the graduated cylinder are shown in Fig. 6.

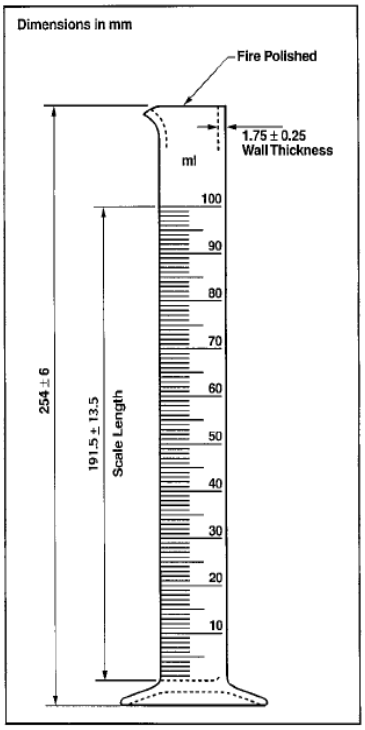
For automated apparatus, the cylinder shall conform to the physical specifications described in the Fig. 6, with the except that graduations below 100 ml mark are permitted as long as they do not interfere with the operation of the level follower. Receiving cylinders for use in automated units may also have a metal base. If required, the receiving cylinder shall be immersed to above the 100 ml graduation line in a cooling liquid contained in a cooling bath, such as, a tall-form beaker of clear glass or placed in a thermostatically controlled aircirculation chamber

6.7.2 *Residue Cylinder*

Of 5 ml or 10 ml capacity, with 0.1 ml graduations beginning at 0.1 ml.

6.8 Temperature Measurement System

6.8.1 Mercury in glass thermometer conforming to the requirements given in Table 1. For partly or fully automatic apparatus and procedure, an alternative means of measuring or recording temperature may also be used provided this gives the same reading under distillation test conditions and has a precision not less than that of the prescribed thermometer.



NOTE—1 to 100 mL in 1 mL graduations; tolerance ± 1.0 mL

FIG. 6 100 ML GRADUATED CYLINDER

Table 1 Thermometer Specifications

(*Clause* 6.8.1)

	Low Range	High Range
Range, °C	-2 to +300	-2 to +400
Graduation, °C	1	1
Immersion, mm	Total	Total
Overall length, mm	381 to 391	381 to 391
Stem diameter, mm	6 to 7	6 to 7
Bulb shape	Cylindrical	Cylindrical
Bulb length, mm	10 to 15	10 to 15
Bulb diameter,	5 to 6	5 to 6
Distance of bottom of bulb to:		
0°C line, mm	100 to 110	25 to 45
300°C line, mm	333 to 354	—
400°C line, mm	—	333 to 354
Longer lines at each, °C	5	5
Numbering at each, °C	10	10
Scale error, °C, *Max*	0.5 to 300	1.0 to 370
Maximum line width, mm	0.23	0.23
Expansion chamber	Required[1)]	—
Heat stability	*see* Note [2)]	*see* Note [2)]

NOTES:

1 An expansion chamber is provided for relief of gas pressure to avoid distortion of the bulb at higher temperatures. It is not for the purpose of joining mercury separations. Under no circumstances shall the thermometer be heated above its highest temperature reading.

2 CAUTION — Under certain test conditions, the bulb of the thermometer can be 28°C above the temperature indicated, and at an indicated temperature of 371°C, the temperature of the bulb is approaching a critical range in the glass. It is thus strongly recommended that distillation temperature readings above 371°C be avoided, but in those cases where thermometers have been exposed to observed temperature readings above 371°C, they shall not be re-used without checking their ice point to verify calibration.

6.8.2 *Electronic Temperature Measurement Devices*

If used, shall exhibit the same temperature lag, emergent-stem effect and accuracy as the equivalent mercury-in-glass thermometer. To simulate the temperature lag of a mercury in-glass thermometer, the circuitry and/or the algorithms used for the electronic system shall take this provision into account.

Alternatively, place the sensor in a casing with the tip covered, so that the assembly, because of its adjusted thermal mass and conductivity, has a temperature lag time similar to that of a mercury in-glass thermometer.

In case of dispute, and unless otherwise agreed, the referee test shall be carried out using the specified mercury-in-glass thermometers.

A means of determining the difference in lag time between an electronic temperature measurement system and a mercury-in-glass thermometer is given in Annex A.

6.9 Centering Device

The temperature sensor shall be fitted through a snug-fitting device designed to mechanically centre the sensor in the neck of the distillation flask without vapour leakage. Examples of acceptable centering devices are shown in Fig. 7 and 8.

Centering devices different from those shown in Fig. 7 and 8 are also acceptable provided that they position and hold the temperature sensor or thermometer in the middle of the neck of the distillation flask as shown in Fig. 9 and 10.

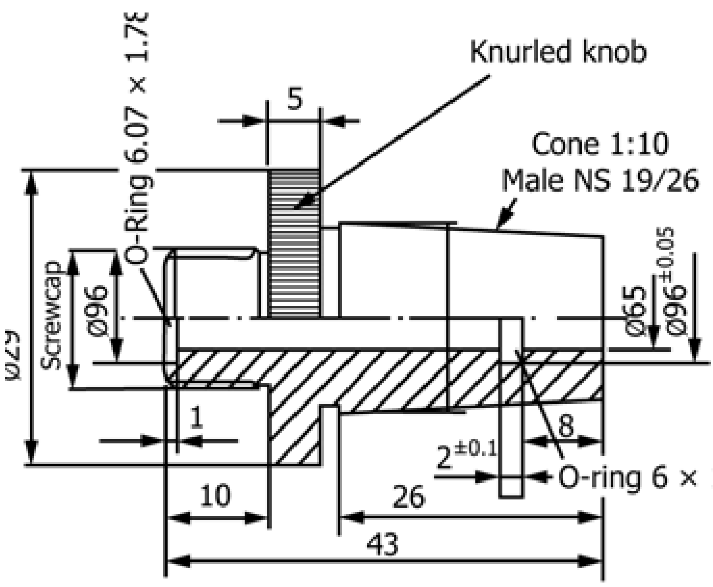
NOTE — When running tests by the manual method, products with a low initial boiling point may have one or more temperature readings obscured by the centering device.

6.10 Barometer

The barometer shall be capable of measuring atmospheric pressure with an accuracy of 0.1 kPa (1 mm Hg) or better, at the same elevation relative to sea level as the apparatus in the laboratory.

Warning — Do not take readings from aneroid barometers that are pre-corrected to give sea level pressures.

NOTE — The barometer should ideally be located in the room in which the distillation is carried out.



Dimensions in mm

FIG. 7 PTFE CENTERING DEVICE FOR GROUND GLASS JOINT

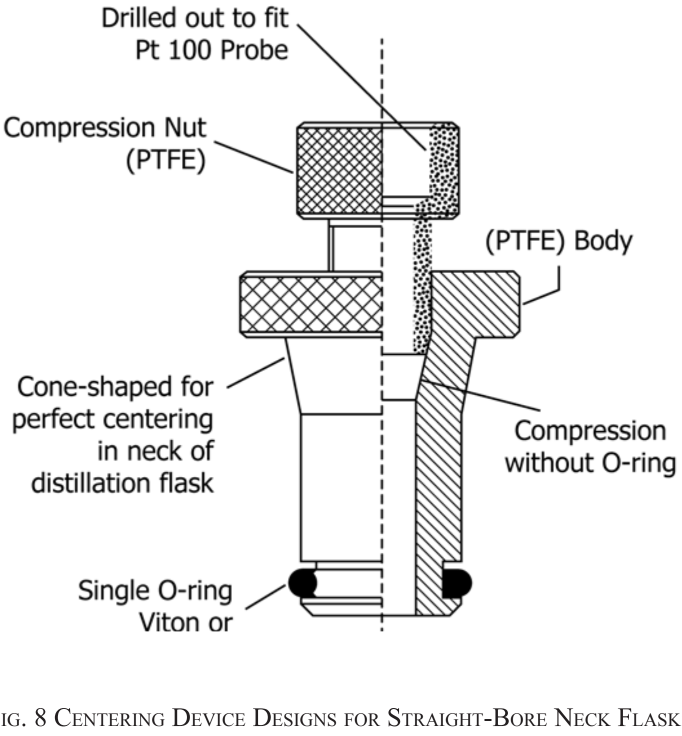


FIG. 8 CENTERING DEVICE DESIGNS FOR STRAIGHT-BORE NECK FLASKS

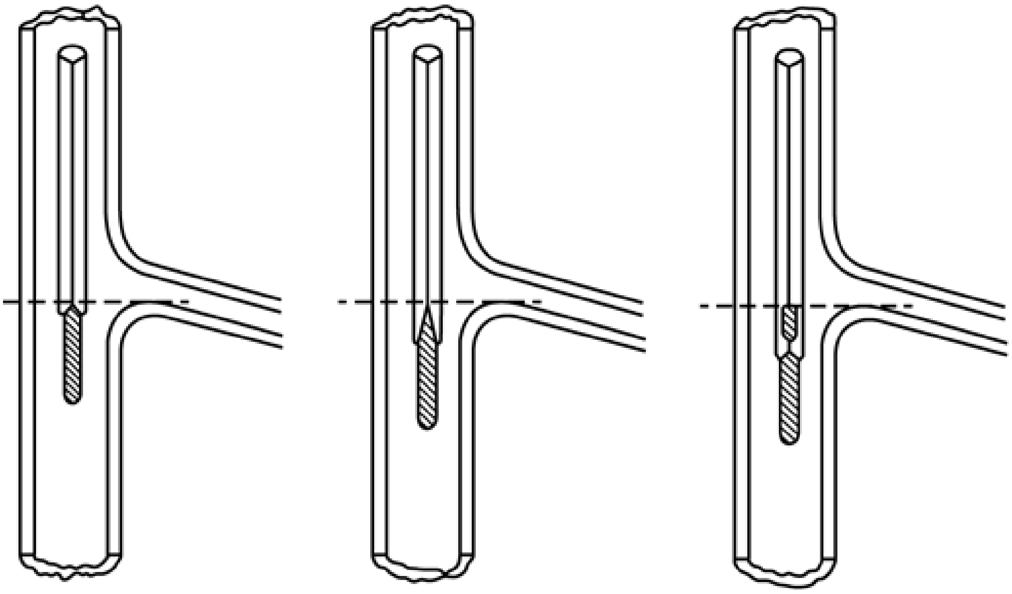


FIG. 9 POSITION OF THERMOMETER IN DISTILLATION FLASK

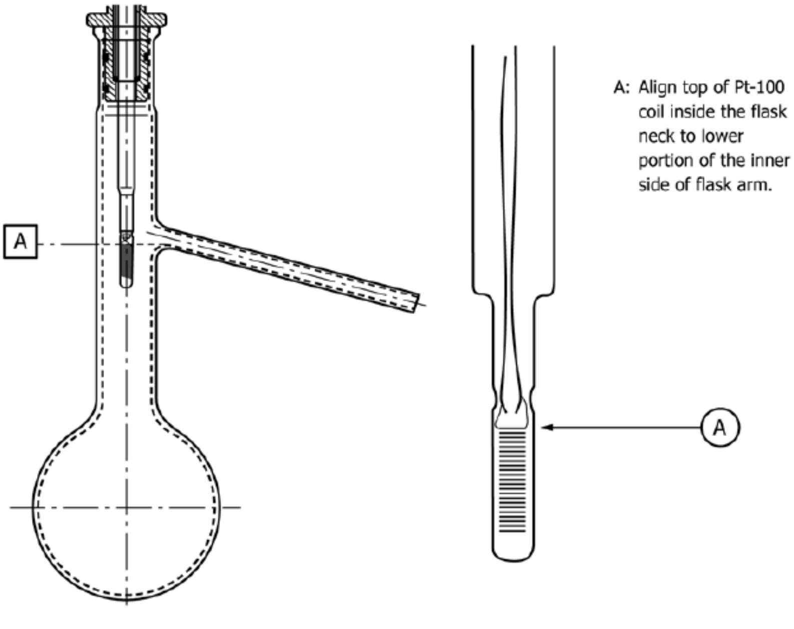

A
A: Align top of Pt-100 coil inside the flask neck to lower portion of the inner side of flask arm.
A

FIG. 10 PLACEMENT OF PT - 100 PROBE RELATIVE TO DISTILLATION FLASK SIDE ARM FOR AUTOMATED DISTILLATION

7 SAMPLE

7.1 Sample Grouping

Determine the nature of the product to be sampled and place it in the appropriate group according to Table 2, which also gives general guidance on sampling conditions.

7.2 Sample Maintenance Prior to Testing

7.2.1 Unless otherwise specified, sampling shall be carried out in accordance with IS 1447 (Part 1) or IS 1447 (Part 2) or ISO 3170 or ISO 3171, based on the special conditions described in Table 2. Maintain samples prior to testing at the specified temperatures given in Table 2, away from sources of direct heat or sunlight.

Table 2 Sample Groups and Sampling Conditions

(*Clauses* 7.1, 7.2.1 and 10.1)

Group number	1	2	3	4
Typical sample type	Gasoline	Gasoline	Wide-cut aviation fuel	Kerosene/ gas oil
Reid vapour pressure, kPa	≥ 65.5	< 65.5	< 65.5	< 65.5
Distillation				
Initial boiling point (IBP), °C	—	—	≤ 100	> 100
Final boiling point (FBP), °C	≤ 250	≤ 250	> 250	> 250
Temperature of sample bottle, °C	< 10	—	—	—
Temperature of sample at sampling, °C	≤ 10	≤ 10	Ambient[1]	Ambient[1]
Temperature of stored sample, °C	< 10[2]	< 10[2]	Ambient	Ambient
If sample is wet (*see* **6.3**)	Resample or dry	Resample or dry	Dry	Dry

NOTES:

1 Samples shall always be at 9°C to 21°C above their pour point, if not fluid at ambient temperature.

2 When no facilities are available for storage below 10°C, the sample may be stored at below 20°C provided the container is tightly sealed (*see* **6.2.2**).

7.2.2 *Groups 1 and 2*

Collect the sample in a container previously cooled to below 10°C. Condition the container, preferably by immersing it in the liquid, where possible, and discarding the first sample. Where immersion is not possible, the sample shall be drawn off into the previously cooled container so that agitation is kept at a minimum. Close the container immediately with a tight-fitting stopper, and place the sample in an ice bath or refrigerator to maintain the sample below the specified temperature. Maintain the sample below 10°C prior to testing, and preferably store at or below this temperature. Where maintenance and/or storage at below 10°C is not possible or practicable, a temperature of up to 20°C is acceptable provided that the sample is always conditioned to a temperature below 10°C before the container is opened.

7.2.3 *Groups 3 and 4*

Maintain the sample at ambient temperature. If the sample is not fluid at ambient temperature, maintain it at a temperature of 9°C to 21°C above its pour point. Shake the sample vigorously prior to sub sampling to ensure homogeneity, and disregard the temperature range shown in Table 3 for the receiving cylinder. Prior to analysis, heat the receiving cylinder to approximately the same temperature as the sample, and pour the heated test portion precisely to the 100 ml mark. Transfer the test portion as rapidly and completely as possible to the distillation flask.

CAUTION — A tightly sealed, full, cold container of sample is likely to break if heated.

7.3 Removing Water from Sample

7.3.1 Samples of materials that are visibly hazy (suspended water) or are suspected of containing water are not suitable for testing.

7.3.2 *Groups 1 and 2*

If the sample is not dry, obtain another sample for testing that is free from suspended water. If such a sample cannot be obtained, add a sufficient amount of anhydrous sodium sulfate or other suitable drying agent to the sample maintained at 0°C to 10°C, and physically remove the water by shaking. Once the sample shows no visible signs of water, use a decanted portion of the sample, maintained at 0°C to 10°C, for the analysis. Record the fact that the sample has been dried by desiccant.

NOTE — Data from a round-robin exercise show that suspended water in hazy samples in groups 1 and 2 can be removed by the above procedure without statistically affecting the results of the test.

7.3.3 *Groups 3 and 4*

In cases where a water free sample is not practical, remove the suspended water by shaking the sample with anhydrous sodium sulfate or another suitable drying agent, and separate it from the drying agent by decantation. Note in the report that the sample has been dried by addition of as desiccant.

8 PREPARATION OF APPARATUS

8.1 Prepare the apparatus by choosing the appropriate distillation flask (125ml), temperature-measurement system and flask support board as required for the indicated group (*see* Table 3). Bring the temperature of the receiving cylinder, the flask, the temperature sensor and the cooling bath to the indicated temperatures.

8.2 Make any necessary provisions so that the temperature of the cooling bath and receiving cylinder are maintained at their specified temperatures. The receiving cylinder shall be positioned in a bath such that either the liquid level is at least as high as the 100 ml mark or the entire receiving cylinder is surrounded by an air circulation chamber

For groups 1, 2 and 3, use suitable media for low-temperature baths include, but not limited to, chopped ice and water, refrigerated brine and refrigerated ethylene glycol.

Table 3 Preparation of Apparatus

(*Clauses* 6.6.1, 7.2.3 and 8.4)

Group number	1	2	3	4
Temperature-measurement system (*see* **5.8**)	Low-range	Low-range	Low-range	High- range
Diameter of hole in flask-support board, mm	38	38	50	50
Temperature at start of test, °C				
a) Flask and thermometer	13 to 18	13 to 18	13 to 18	≤ ambient
b) Flask support board and shield	≤ ambient	≤ ambient	≤ ambient	—
c) Receiving cylinder and sample	13 to 18	13 to 18	13 to 18	13 to ambient

For group 4, use suitable media for ambient and higher bath temperatures include, but not limited to, cold water, hot water and heated ethylene glycol.

8.3 Remove any residual liquid in the condenser tube by swabbing with a piece of soft, lint-free cloth attached to a cord or wire.

8.4 For samples in groups 1, 2 and 3, fit a low range thermometer, provided with a snug fitting cork or silicone rubber stopper, tightly into the neck of the sample container, and bring the temperature of the sample to that specified in Table 3.

8.5 When the temperature of the sample is confirmed, measure the test portion precisely to the 100 ml mark of the receiving cylinder, and then transfer it as completely as practical to the distillation flask, taking care that none of the liquid flows into the vapour tube.

NOTE — Any material which evaporates during the transfer will contribute to the loss; any material which remains in the receiving cylinder will contribute to the observed recovery volume at the time of the initial boiling point.

If irregular boiling (bumping) is expected, add a small volume of clean and dry boiling chips to the test portion.

NOTE — Although, theoretically, the volume of deposit on the chips is part of the residue, this volume is negligible and can be disregarded.

8.6 Fit the appropriate temperature sensor through a centering device (**6.9**). In the case of a mercury-in-glass thermometer, the bulb is centered in the neck and the lower end of the capillary is level with the highest point on the bottom of the inner wall of the vapour tube (*see* Fig. 8). In the case of a thermocouple/resistance thermometer, follow the manufacturer's instructions as to placement so that the sensing position is just below the level of the side arm of the distillation flask. Use a small quantity of vacuum grease on the mating surface of the PTFE centering device to facilitate dismantling after use.

8.7 Fit the flask vapour tube, provided with a snug-fitting, well-rolled cork or silicone rubber stopper, tightly into the condenser tube. Adjust the distillation flask in a vertical position so that the vapour tube extends into the condenser tube for a distance of 25 mm to 50 mm. Raise and adjust the flask-support board to fit snugly against the bottom of the flask.

8.8 Place the receiving cylinder that was used to measure the test portion, without drying, fitted with a drip deflector if necessary, into the bath under the lower end of the condenser tube so that the end of the condenser tube is centered in the receiving cylinder and extends therein for a distance of at least 25 mm, but not below the 100 ml mark. For manual distillations, cover the receiving cylinder closely with a piece of blotting paper or similar material that has been cut to fit the condenser tube snugly. For automated distillations, cover the receiving cylinder, if necessary, with the device provided with the apparatus.

8.9 If receiver deflector is not used keep the drip tip of the condenser away from the wall of the receiving cylinder. Note the start time. Observe and record the IBP to the nearest 0.5^{0}C. If a receiver deflector is not being used, immediately move the receiving cylinder so that the tip of the condenser touches its inner wall.

8.10 Record the room temperature and prevailing barometric pressure. Proceed at once with the distillation in accordance with **9** or **10**, depending on whether the procedure is manual or automatic.

9 APPARATUS VERIFICATION

9.1 Level Follower

For automated apparatus, the level follower/ recording mechanism of the apparatus shall have a resolution of 0.1 ml with a maximum error of 0.3 ml between the 5 ml and 100 ml points. The calibration of the assembly shall be verified according to the manufacturer's instructions at intervals of not more than three months and after the system has been replaced or repaired.

NOTE — The typical verification procedure involves verifying the output with the receiving cylinder containing 5 ml and 100 ml of material respectively.

9.2 Electronic Temperature Measurement Devices

9.2.1 Temperature measurement devices using other than the specified mercury-in-glass thermometers shall exhibit the same temperature lag, emergent-stem effect and accuracy as the equivalent mercury-in-glass thermometer. Verification of the calibration of these temperature sensors shall be carried out at intervals of not more than six months and after the system has been replaced or repaired. The electronic circuit for resistance thermometers shall be verified against a standard precision resistance. When performing this verification, ensure that any algorithms used to correct the temperature for lag and emergent stem effect are not applied (refer the manufacturer's instructions). The response of the temperature measurement system itself shall also be verified by one of the procedures described in **9.2.2** or **9.2.3**.

9.2.2 Distil reagent grade toluene in accordance with group I of this test method and compare the 50 percent *(V/V)* recovered temperature with that shown by the above-mentioned mercury-in-glass thermometer when carrying out a manual test under the same conditions. Use reagent grade toluene for this test unless it can be shown that the grade proposed is of sufficient purity to permit its use without lessening the accuracy of the determination.

Toluene is used as a verification fluid for bias; it will yield almost no information on how well an electronic measurement system simulates the temperature lag of a mercury-in-glass thermometer. Annex A describes a method for determining this difference in lag time.

To verify the response at elevated temperatures, use hexadecane.

NOTE — Toluene is shown in reference manuals as boiling at 110.6°C, and hexadecane at 287.0°C, under the conditions specified in ISO 918, which uses a partial immersion thermometer. Because this Standard uses thermometers calibrated for total immersion, the results will be lower and different for each. The approximate values obtained from an inter-laboratory study are shown below:

	Manual Apparatus	**Automated Apparatus**
Toluene (groups 1, 2, 3)	105.9-111.8°C	108.5-109.7°C
Hexadecane (group 4)	272.2-283.1°C	277.0-280.0°C

9.2.3 Distil a certified reference fluid of a specific product group, under the manual conditions, using in sequence the appropriate mercury-in-glass thermometer followed by the alternative temperature measurement device or system. Record systematic deviations over the range of temperatures measured to ensure that no deviation exceeds the repeatability of this test method, and that the algebraic sum of the deviations is close to zero.

NOTE — Certified reference fluids, consisting of specified mixtures of single compounds, are commercially available for group 1 and group 4 product types.

9.3 Electronic Pressure Measuring Device

At intervals of not more than six months, and after the system has been repaired or replaced, the barometric reading of the device shall be verified against a barometer as specified in **6.10**.

10 PROCEDURE FOR MANUAL APPARATUS

10.1 Record the prevailing barometric pressure. Ensure that the sample is conditioned in accordance with Table 2.

10.2 Apply heat to the distillation flask (**6.2**) and contents, with the drip tip of the condenser (**6.3**) away from the wall of the receiving cylinder (**6.7.1**) allowing observation for the first condensation. Regulate the heating (**6.5**) at this stage so that the time interval between the first application of heat and the initial boiling point is as specified in Table 4.

10.3 Observe and record the first drop of condensate as the initial boiling point to the nearest 1°C. If a receiver deflector is not used, immediately move the receiving cylinder so that the tip of the condenser touches the inner wall.

10.4 Regulate the heating so that the time from initial boiling point to 5 percent (*V/V*) recovered is as indicated in Table 4.

Table 4 Test Conditions

[*Clause* 10.2, 10.4, 10.14 (c) *and* 11.1]

Group number	**1**	**2**	**3**	**4**
Temperature of condenser bath, °C	0 to 1	0 to 4	0 to 4	0 to 60
Temperature of medium around receiving cylinder, °C	13 to 18	13 to 18	13 to 18	± 3 of charge
Time from first heat to IBP, min	5 to 10	5 to 10	5 to 10	5 to 15
Time from IBP to: 5percent *(V/V)* recovered, s	60 to 100	60 to 100	—	—
Uniform average rate from 5 percent (V/V) recovered to 5 ml in flask, ml/min	4 to 5	4 to 5	4 to 5	4 to 5
Time from 5 ml residue in flask to FBP, min	≤5	≤5	≤5	≤5

10.5 Continue to regulate the heating so that the uniform average rate of condensation from 5 percent (*V/V*) recovered to 5 ml residue in the flask is 4 ml/min to 5 ml/min.

Due to the configuration of the distillation flask and the conditions of the test, the vapour and liquid around the temperature sensor (**6.8**) are not in thermodynamic equilibrium. The distillation rate will consequently have an effect on measured vapour temperature and thus should be kept as constant as possible throughout the test.

NOTE — When testing group 1 or group 2 materials, the condensate may form non-miscible phases and bead upon the thermometer and in the neck of the distillation flask at a vapour temperature of approximately 160°C. This is accompanied by a sharp (approximately 3°C) dip in vapour temperature and a drop in recovery rate. The phenomenon may last for 10 s to 20 s before the temperature recovers and the condensate starts flowing again. This point may be referred to as the "hesitation point".

10.6 Repeat any distillation that does not meet the conditions described in **10.1** to **10.4**.

10.7 If a decomposition point is observed, discontinue the heating and proceed according to **10.10**.

10.8 In the interval between the initial boiling point and the end of the distillation, observe and record data necessary for the calculation and reporting of the test results as required by the particular specification or as previously established for the material under test.

NOTE — These observed data may include thermometer readings at specified percent recovered or percent recovered at specified thermometer readings.

10.9 Record all volumes in the receiving cylinder to the nearest 0.5 ml and all thermometer readings to the nearest 0.5°C.

a) For groups 1, 2, 3 and 4, in cases in which no specific data requirements have been indicated, record the initial boiling point, the final boiling point or dry point, or both, and thermometer readings at 5 percent and 95 percent volume recovered, and at each 10 percent multiple of volume recovered from 10 percent to 90 percent inclusive.

b) For group 4, when the high range thermometer issued for testing kerosene, pertinent thermometer readings can be obscured by the centering device. If these readings are required, perform a second distillation in accordance with the requirements of group 3. In such cases, readings from the low range thermometer are reported in place of the obscured high-range thermometer readings, and the test report shall indicate this. If, by agreement, the obscured readings are waived, the test report shall also indicate this.

10.10 When required to report the thermometer reading at a specified percent evaporated or percent recovered for a sample which has a rapidly changing distillation curve slope in the region of the specified percent evaporated or percent recovered reading, record thermometer readings at every 1 percent (*V/V*) recovered. The slope is considered to be rapidly changing if the change in the slope, C, of the data points described above for groups 1, 2, 3 and 4 in that particular area is greater than 0.6 as calculated from Equation (1):

Change of Slope (C) =

$$(C_2 - C_1)/(V_2 - V_1) - (C_3 - C_2)/(V_3 - V_2) \quad \text{------ (1)}$$

Where,

C_1 = temperature at the volume percent recorded one reading, prior to the volume percent in question, °C,

C_2 = temperature at the volume percent recorded in question, °C,

C_3 = temperature at the volume percent recorded following the volume percent in question,°C,

V_1 = Volume percent recorded one reading prior to the volume percent,

V_2 = Volume percent recorded at the volume percent in question, and

V_3= Volume percent recorded following the volume percent in question.

10.11 Since it is extremely difficult to determine the amount of boiling residual liquid in the distillation flask, this is estimated from the amount of liquid recovered in the receiving cylinder at this time. The dynamic hold-up has been determined to be approximately 1.5 ml at this point, and thus with no losses, the amount of 5 ml in the distillation flask can be assumed to correspond to an amount of 93.5 ml in the receiving cylinder.

If the actual loss differs by more than 2 ml from the estimated value, repeat the test.

10.12 Observe and record the final boiling point, or dry point, or both, as required, and discontinue heating.

The end point (final boiling point), rather than that is, dry point is intended for general use. The dry point can be reported in connection with special purpose naphtha, such as, those used in paint industry. Also, it is substituted for the end point (final boiling point) whenever the sample is of such a nature that the precision of the end point (final boiling point) cannot consistently meet the requirements given in the precision section.

10.13 Allow the distillate to continue to drain into the receiving cylinder, observe and note the volume of condensate to the nearest 0.5 ml, at 2 min intervals, until two successive observations agree. Measure this volume accurately and record it to the nearest 0.5 ml as the percent recovery. If the distillation was previously discontinued under the conditions of a decomposition point, deduct the percent recovery from 100, report this difference as the percent residue and percent loss, and omit the procedure according to **10.11**.

10.14 After the distillation flask has cooled and no more vapours are observed, disconnect the distillation flask from the condenser, pour its contents into the 5 ml residue cylinder (**6.7.2**) and, with the distillation flask suspended over the 5 ml residue cylinder, allow the flask to drain until no appreciable increase in volume of the liquid in the 5 ml residue cylinder is observed. Record the volume in the residue cylinder to the nearest 0.1 ml as the percent residue.

a) If boiling chips were added, care should be taken to avoid their inclusion in this volume.
b) If the 5 ml residue cylinder does not have graduations below 1 ml, and the volume of liquid is less than 1 ml, pre-fill the residue cylinder with 1 ml of heavy oil to obtain a better reading of the volume of residue.
c) For group 4 products, examine the condenser arm and the side arm of the distillation flask for waxy or solid deposits. If found, repeat the test, adjusting the temperature of the condenser bath upwards within the range specified in Table 4.

11 PROCEDURE FOR AUTOMATED APPARATUS

11.1 Apply heat to the distillation flask (**6.2**) and its contents. Ensure that the drip deflector tip is touching the wall of the receiving cylinder. Regulate the heating (**6.5**) at this stage so that the time interval between the first application of heat and the initial boiling point is as specified in Table 4.

11.2 Record the first drop of condensate as the initial boiling point to the nearest 0.1 °C.

11.3 Follow the procedure mentioned in points **10.3** to **10.6**.

11.4 Record all volumes in the receiving cylinder to the nearest 0.1 ml and all temperature readings to the nearest 0.1 °C.

In cases in which no specific data requirements have been indicated, record the initial boiling point, the final boiling point or dry point, or both, and thermometer readings at 5 percent and 95 percent volume recovered, and at each 10 percent multiple of volume recovered from 10 percent to 90 percent inclusive.

11.5 When the residual liquid in the distillation flask is approximately 5 ml, follow the procedure mentioned in point **10.8** to **10.11.**

If the actual loss differs by more than 2 ml from the estimated value, repeat the test.

12 CALCULATIONS

12.1 The percent total recovery is the sum of the percent recovery (*see* **10.10**) and the percent residue (*see* **10.11**). Deduct the percent total recovery from 100 to obtain the percent loss.

12.2 Do not correct the barometric pressure for meniscus depression, and do not adjust the pressure to what it would beat sea level.

NOTE — The observed barometric reading does not have to be corrected to a standard temperature and to standard gravity. Even without performing these corrections, the corrected temperature readings for the same sample between laboratories at two different locations in the world will, in general, differ less than 0.1°C at 100°C. Almost all data obtained earlier have been reported at barometric pressures that have not been corrected to standard temperature and to standard gravity.

12.3 Correct temperature readings to 101.3 kPa (760 mm Hg) pressure. Obtain the correction to be applied to each temperature reading by means of the Sydney Young equations given in equation 2 or equation 3, as appropriate, or by the use of Table 5. For celsius temperatures:

$$Cc = 0.0009\ (101.3 - P_k)(273 + tc) \quad \text{----------}(2)$$

$$Cc = 0.00012\ (760 - P)(273 + tc) \quad \text{----------}\ (3)$$

Where,

tc = the observed temperature reading, in °C;

Cc = corrections to be added algebraically to the observed temperature readings;

P_k = barometric pressure, prevailing at the time and location of the test, kPa; and

P = barometric pressure, prevailing at the time and location of the test, mm Hg.

After applying the corrections and rounding each result to the nearest 0.5°C or 0.1°C, as appropriate to the apparatus being used, use the corrected temperature readings in all further calculations and reporting.

NOTE — Temperature readings are not corrected to 101.3 kPa (760mm Hg) when product definitions, specifications, or agreements between the parties involved indicate, specifically, that such correction is not required or that correction shall be made to some other base pressure.

Table 5 Approximate Thermometer Reading Correction

(*Clause* 12.3)

Temperature Range		Correction[A] per 1.3 kPa (10 mm Hg) Difference in Pressure	
°C	°F	°C	°F
10-30	50-86	0.35	0.63
30-50	86-122	0.38	0.68
50-70	122-158	0.40	0.72
70-90	158-194	0.42	0.76
90-110	194-230	0.45	0.81
110-130	230-266	0.47	0.85
130-150	266-302	0.50	0.89
150-170	302-338	0.52	0.94
170-190	338-374	0.54	0.98
190-210	374-410	0.57	1.02
210-230	410-446	0.59	1.07
230-250	446-482	0.62	1.11
250-270	482-518	0.64	1.15
270-290	518-554	0.66	1.20
290-310	554-590	0.69	1.24
310-330	590-626	0.71	1.28
330-350	626-662	0.74	1.33
350-370	662-698	0.76	1.37
370-390	698-734	0.78	1.41
390-410	734-770	0.81	1.46

[A] Values to be added when barometric pressure is below 101.3 kPa (760 mm Hg) and to be subtracted when barometric pressure is above 101.3 kPa.

12.4 Correct the actual loss to 101.3 kPa (760 mm Hg) pressure when temperature readings are corrected to 101.3 kPa pressure. The corrected loss, *Lc*, is calculated from equation 4 or equation 5, as appropriate

$$L_c = 0.5+(L-0.5)/\{1+(101.3-P_k)/8.00 \quad \text{---------} \ (4)$$

$$L_c = 0.5+(L-0.5)/\{1+(760 - P)/60.0\} \quad \text{---------} \ (5)$$

Where,

L = observed loss,

L_c = corrected loss,

P_k = pressure, kPa, and

P = pressure, mm Hg.

NOTE — Equation 4 and 5above have been derived from D 86 – 16a

12.4.1 Calculate the corresponding corrected percent recovery in accordance with the following equation:

$$R_c = R + (L-L_c) \quad \text{-----------------} \ (6)$$

Where,

L = percent loss or observed loss,

L_c = corrected loss, in percent volume,

R = percent recovery, and

Rc = corrected percent recovery

12.5 To obtain the percent evaporated at a prescribed temperature reading, add the percent loss to each of the observed percent recovered at the prescribed temperature readings, and report these results as the respective percent evaporated, that is:

$$P_e = P_r + L \quad \text{----------------} \ (7)$$

Where,

L = observed loss, in percent volume;

P_e = percent evaporated; and

P_r = percent recovered.

12.6 To obtain temperature readings at prescribed percent evaporated, and if no recorded temperature data is available within 0.1 volume percent of the prescribed percent evaporated, use either of the two following procedures, and indicate on the report whether the arithmetical procedure or the graphical procedure has been used.

12.6.1 *Arithmetical Procedure*

Deduct the observed loss from each prescribed percent evaporated to obtain the corresponding percent recovered. Calculate each required temperature reading as follows:

$$T = T_L + (T_H - T_L)(P_r - P_{rl})/(P_{rH} - P_{rL}) \quad \text{---------} \ (8)$$

Where,

P_r = percent recovered corresponding to the prescribed percent evaporated;

P_{rH} = percent recovered adjacent to, and higher than P_r;

P_{rL} = percent recovered adjacent to, and lower than P_r;

T = temperature reading at the prescribed percent evaporated;

T_H = temperature reading recorded at P_{rH}; and

T_L = temperature reading recorded at P_{rL}.

Values obtained by the arithmetical procedure are affected by the extent to which the distillation graphs are nonlinear. Intervals between successive data points can, at any stage of the test, be no wider than the intervals indicated in **10.10**. In no case shall a calculation be made that involves extrapolation.

12.6.2 *Graphical Procedure*

Using graph paper with uniform subdivisions, plot each temperature reading corrected for barometric pressure, if required (*see* **12.2**), against its corresponding percent recovered. Plot the IBP at 0 percent recovered. Draw a smooth curve connecting the points. For each prescribed percent evaporated, deduct the distillation loss to obtain the corresponding percent recovered and take from the graph the temperature reading that this percent recovered indicates. Values obtained by graphical interpolation procedures are affected by the care with which the plot is made.

12.6.3 In most automated instruments, temperature-volume data are collected at 0.1 volume percent intervals or less and stored in memory. To report a temperature reading at a prescribed percent evaporated, neither of the procedures described in **12.6** have to be used. Obtain the desired temperature directly from the database as the temperature closest to and within 0.1 volume percent of the prescribed percent evaporated.

13 EXPRESSION OF RESULTS

13.1 Report the sampling procedure used.

13.2 Report all volume percentages to the nearest 0.5 percent (*V/V*) for the manual procedure or to the nearest 0.1 percent (*V/V*) for the automated procedure and record all temperature readings to the nearest 0.5°C for the manual procedure or to the nearest 0.1 °C for the automated procedure, as appropriate for the apparatus being used.

13.3 Report whether the manual or automated procedure has been used.

13.4 Unless specifically requested (*see* Note below **12.2**), temperature readings shall be corrected to a barometric pressure of 1013 kPa before being reported. When such special arrangements have been made, report the observed values in accordance with **13.2**, together with the laboratory barometric pressure. In these cases, also report the residue and loss as "observed".

13.5 Report samples in groups 1 and 2 as "dried" if the default procedure according to **7.3.2** has been carried out.

13.6 Report the substitution of a low-range thermometer for the high-range thermometer if the conditions of **10.9 ii)** are applied.

14 PRECISION - MANUAL APPARATUS

14.1 Repeatability

14.1.1 *Group 1*

The difference between two test results obtained by the same operator, using the same apparatus under constant operating conditions on identical test material, would, in the long run, and with normal and correct application of the test method, exceed the values given in Table 7 in only one case in 20.

14.1.2 *Groups 2, 3 and 4*

The difference between two test results obtained by the same operator, using the same apparatus under constant operating conditions on identical test material, would, in the long run and with normal and correct application of the test method, exceed the values given in Table 8 in only one case in 20.

14.2 Reproducibility

14.2.1 *Group 1*

The difference between two single and independent test results obtained by different operators working in different laboratories on identical test material would, in the long run, and with normal and correct application of the test method, exceed the values given in Table 7 in only one case in 20.

14.2.2 *Groups 2, 3 and 4*—The difference between two single and independent test results obtained by different operators working in different laboratories on identical test material would, in the long run, and with normal and correct operation of the test method, exceed the values given in Table 8 in only one case in 20.

14.3 Slope or Rate of Change of Temperature

14.3.1 The precision of the IBP and EP does not require any slope calculation. The slope at any point during the distillation is calculated from the following equation, using the values shown in Table 6.

$$S_c \ (or\ S_F) = (Tu - T_L) \ / \ Vu - V_L) \qquad \text{---------} \ (9)$$

Where,

S_c = the slope, °C/volume percent,

S_F = the slope, °F/volume percent,

Tu = the upper temperature °C(or °F),

T_L = the lower temperature °C(or °F),

Vu = the volume percent recovered or evaporated corresponding to Tu, and

V_L = the volume percent recovered or evaporated corresponding to T_L.

14.3.2 In the event that the distillation end point occurs prior to the 95 percent point, the slope at the end point is calculated as follows:

$$Sc \ (\text{or } S_F) = (T_{EP} - T_{HR})/(V_{EP} - V_{HR}) \quad \text{---------} \ (10)$$

Where,

T_{EP} or T_{HR} = the temperature, in °*C* or °*F,* at the percent volume recovered indicated by the subscript, and

V_{EP} or V_{HR} = the volume percent recovered,

Subscript EP = end point, and

Subscript HR = highest reading, either 80 percent or 90 percent, prior to the end point.

Table 6 Data Points for Determining Slope, S_C or S_F

(*Clause* 14.3.1)

Slope at percent	IBP	5	10	20	30	40	50	60	70	80	90	95	EP
T_L at percent	0	0	0	10	20	30	40	50	60	70	80	90	95
T_U at percent	5	10	20	30	40	50	60	70	80	90	90	95	V_{EP}
V_U - V_L	5	10	20	20	20	20	20	20	20	20	10	5	V_{EP}-95

14.3.3 For points between 10 percent to 85 percent recovered, the slope is calculated as follows:

$$Sc \text{ (or } S_F) = 0.05\ (T_{(V+10)} - T_{(V-10)}) \text{ ---------- } (11)$$

Table 7 Repeatability and Reproducibility for Group 1

(*Clause* 14.1.1 and 14.2.1)

Evaporated Point, Percentage	Manual Repeatability		Manual Reproducibility	
	°C	°F	°C	°F
IBP	3.3	6	5.6	10
5	$1.9+0.86S_C$	$3.4+0.86S_F$	$3.1+1.74S_C$	$5.6+1.74S_F$
10	1.2+0. 86S_C	$2.2+0.86S_F$	$2.0+1.74S_C$	$3.6+1.74S_F$
20	1.2+0. 86S_C	$2.2+0.86S_F$	$2.0+1.74S_C$	$3.6+1.74S_F$
30-70	1.2+0. 86S_C	$2.2+0.86S_F$	$2.0+1.74S_C$	$3.6+1.74S_F$
80	1.2+0. 86S_C	$2.2+0.86S_F$	$2.0+1.74S_C$	$3.6+1.74S_F$
90	1.2+0. 86S_C	$2.2+0.86S_F$	$0.8+1.74S_C$	$3.6+1.74S_F$
95FBP	1.2+0. 86S_C	$2.2+0.86S_F$	$1.1+1.74S_C$	$1.9+1.74S_F$
FBP	3.9	7	7.2	13

NOTE — Sc or S_F is the average slope (or rate of change) calculated in accordance with **14.3** Table 8 precision data obtained from RR study on both manual and automated ASTM D86 units by North American and IP laboratories.

Table 8 Repeatability and Reproducibility for Group 2, 3 and 4 (Manual Method)

	Repeatability		Reproducibility	
	°C	°F	°C	°F
IBP	$1.0+0.35S_C$	$1.9+0.35S_F$	$2.8+0.93S_C$	$5.0+0.93S_F$
5-95 percent	$1.0+0.41S_C$	$1.8 +0.41S_F$	$1.8+1.33S_C$	$3.3+1.33S_F$
FBP	$0.7+0.36S_C$	$1.3+0.36S_F$	$3.1+0.42S_C$	$5.7+0.42S_F$
Percent volume at temperature reading	$0.7+0.92/ S_C$	$0.7+1.66/S_F$	$1.5+1.78/S_C$	$1.53+3.20/S_F$

NOTE — S_C or S_F is the average slope (or rate of change) calculated in accordance with **14.3**. Table 9 has been derived from ASTM D86–97

15 PRECISION - AUTOMATED APPARATUS

15.1 Repeatability

15.1.1 *Group 1, 2 and 3*

The difference between two test results obtained by the same operator using the same apparatus under constant operating conditions on identical test material would, in the long run, and with normal and correct application of the test method, exceed the values given in Table 9 in only one case in 20.

15.1.2 *Group 4*

The difference between two test results obtained by the same operator with the same apparatus under constant operating conditions on identical test material would, in the long run, and with normal and correct application of the test method, exceed the values given in Table 10 in only one case in 20.

15.2 Reproducibility

15.2.1 *Group 1, 2 and 3*

The difference between two single and independent test results obtained by different operators working in different laboratories on identical test material would, in the long run, and with normal and correct operation of the test method, exceed the values given in Table 9 in only one case in 20.

15.2.2 *Group 4*

The difference between two single and independent test results obtained by different operators working in different laboratories on identical test material would, in the long run, and with normal and correct application of the test method, exceed the values given in Table 10 in only one case in 20.

Table 9 Repeatability and Reproducibility for Group 1, 2, 3 (Automated) (Valid Range 20°C to 260°C)

(*Clause* 15.2.1)

Percent Evaporated	Repeatability°C	Reproducibility°C
IBP	2.7	4.7
5	$1.4+2.8(0.43S_C + 0.24)$	$2.5 + 2.8(0.43S_C + 0.24)$
10	$0.9+2.8(0.43S_C + 0.24)$	$1.9 + 2.8(0.43S_C + 0.24)$
20	$0.9+2.8(0.43S_C + 0.24)$	$2.0 + 2.8(0.43S_C + 0.24)$

Table 9 *(Concluded)*

Percent Evaporated	Repeatability°C	Reproducibility°C
30	$0.8+2.8(0.43S_C + 0.24)$	$1.8 + 2.8(0.43S_C + 0.24)$
40	$0.9+2.8(0.43S_C + 0.24)$	$2.0 + 2.8(0.43S_C + 0.24)$
50	$1.0+2.8(0.43S_C + 0.24)$	$1.9 + 2.8(0.43S_C + 0.24)$
60	$1.1 + 2.8(0.43S_C + 0.24)$	$2.0 + 2.8(0.43S_C + 0.24)$
70	$1.5 + 2.8(0.43S_C + 0.24)$	$2.1 + 2.8(0.43S_C + 0.24)$
80	$1.1 + 2.8(0.43S_C + 0.24)$	$2.0 + 2.8(0.43S_C + 0.24)$
90	$1.8 + 2.8(0.43S_C + 0.24)$	$2.8 + 2.8(0.43S_C + 0.24)$
95	$2.0 + 2.8(0.43S_C + 0.24)$	$3.6 + 2.8(0.43S_C + 0.24)$
FBP	3.3	7.1

Where,

Sc = slope or rate of change of temperature, in degree celsius.

Table 10 Repeatability and Reproducibility for Group 4 (Automated)

(*Clause* 15.2.2)

Percent Recovered	Repeatability °C	Reproducibility °C	Valid R
IBP	0.018T	0.055T	145 to 220
5 percent	0.0109T	0.03T	160 to 255
10	0.0094T	0.022T	160 to265
20	0.00728T	0.0208T	175 to275
30	0.582T	0.0165T	185 to285
40	0.005T	0.014T	195 to290
50	1.0	3.0	170 to295
60	0.00357T	0.0117T	220 to305
70	0.00355T	0.0125T	230 to315
80	0.00377T	0.0136T	240 to325
90	0.0041T	0.015T	180 to340
95	0.01318 (T-140)	0.04105(T-40)	260 to360
FBP	2.2	7.1	195to365

Where,

T = Percent recovered temperature within valid range prescribed.

Table 11 Precision for Percent Evaporated at a Prescribed Temperature — Gasoline (Consolidated Equation)

Valid Range E70 – E 180°C (Automated Apparatus)		
	r	R
	0.00836 (150-X)	0.0200 (150-X)
where X = percent evaporated in the prescribed temperature		

Table 12 Precision for Percent Recovered at a Prescribed Temperature — Diesel

Valid Range R 200 – R 300°C (Automated Apparatus)		
R 200C, R 250C, R 300C		
	r	R
	1.07	2.66

16 TEST REPORT

The test report shall contain at least the following information:

a) Reference to this standard;

b) Type and complete identification of the product tested;

c) Result of the test (*see* **13**);

d) Any deviation, by agreement or otherwise, from the procedure specified; and

e) Date of the test.

ANNEX A

(*Clauses* 6.8.2 and 9.2.2)

DETERMINATION OF THE DIFFERENCE IN LAG TIME BETWEEN AN ELECTRONIC TEMPERATURE MEASUREMENT SYSTEM AND A MERCURY-IN-GLASS THERMOMETER

A-1 The response time of an electronic temperature measuring device is inherently more rapid than that of a mercury in-glass thermometer. The temperature measuring device assembly in general use, consisting of the sensor and its casing, or an electronic system and its associated software, or both, is so designed that the temperature measuring system will simulate the temperature lag of the mercury-in-glass thermometer.

A-2 To determine the difference in lag time between such a temperature measuring system and a mercury-in-glass thermometer, analyze a sample, such as, gasoline, kerosene, jet fuel, or light diesel fuel with the electronic temperature measurement system in place and in accordance with the procedures described in this test method. In most cases this is the standard distillation step performed with an automated unit.

A-3 Do not use a single pure compound, a very narrow boiling range product, or a synthetic blend of less than six compounds for this test.

A-4 Best results are obtained with a sample that is typical of the sample load of the laboratory. Alternatively, use a full-range mixture with a 5 percent to 95 percent boiling range of at least 100°C.

A-5 Replace the electronic temperature measuring device with a low range or high range mercury-in-glass thermometer, depending on the boiling range of the sample.

A-6 Repeat the distillation with this thermometer, and manually record the temperature at the various percent recovered as described in **11.4**.

A-7 Calculate the values for the repeatability for the observed slope ($\Delta T/\Delta V$) for the different readings in the test.

A-8 Compare the test data obtained using these two temperature measuring devices. The difference at any point shall be equal to, or less than, the repeatability of the method at that point. If this difference is larger, replace the electronic temperature measuring device or adjust the electronics involved, or both.

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